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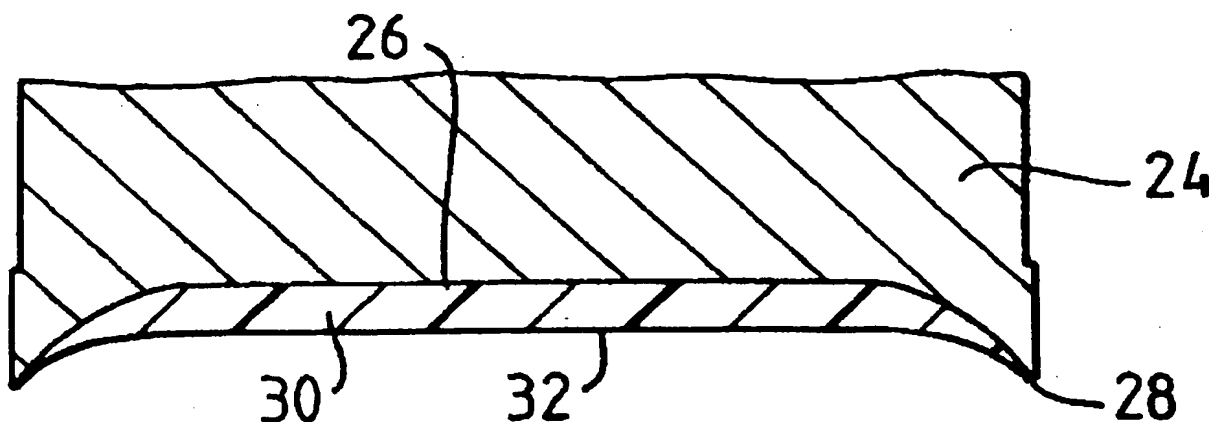
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GB 2260989 A GB 1355521 A GB 0736591 A
EP 0276971 A1

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(54) Process for making shaped articles

(57) A process for the manufacture of articles, (e.g. detergent products) in shaped solid form, comprises compacting a particulate composition is characterised by use of mould parts 24 which have, over at least part of their surface area 32 which contacts the detergent product, a total modulus of elasticity within the range from 10^5 up to $8 \times 10^7 \text{ Nm}^{-2}$. The use of such mould parts reduces unwanted adhesion of the composition to the mould parts. The composition may contain 5 to 60% by weight non-soap detergent, 10 to 70 weight % detergency builder and 25 to 85% by weight of other materials. The desired modulus may be obtained by coating the part of surface of the mould parts with an elastomer.

Fig. 5.



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At least one drawing originally filed was informal and the print reproduced here is taken from a later filed formal copy.

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Fig. 1.

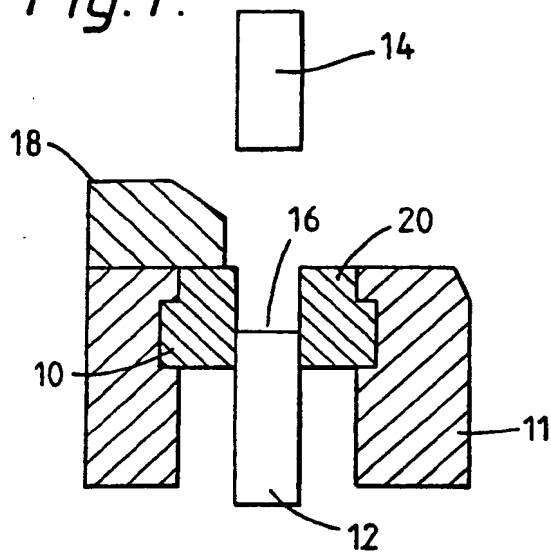
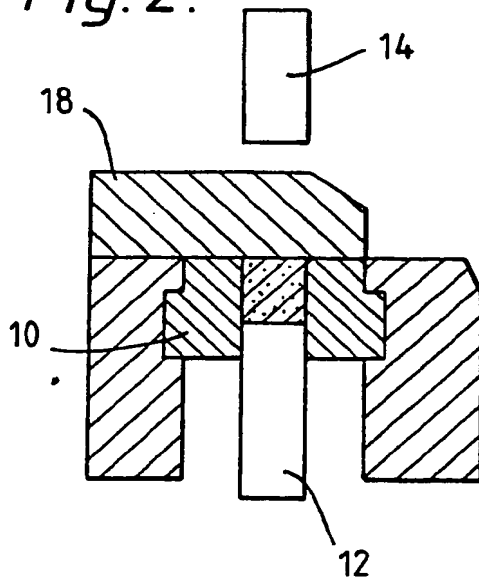


Fig. 2.



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Fig. 3.

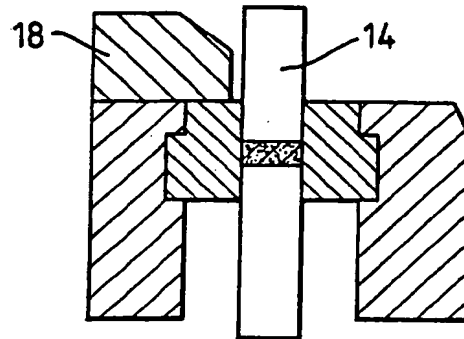


Fig. 4.

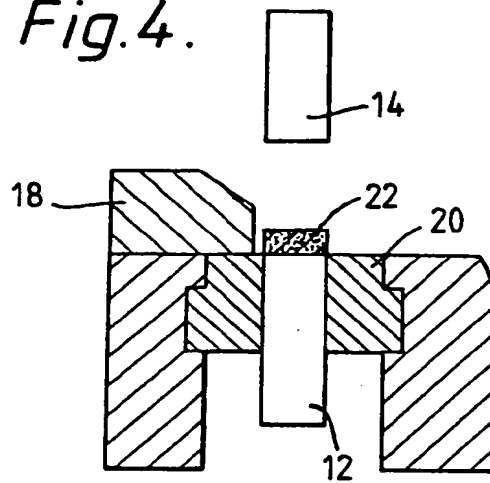
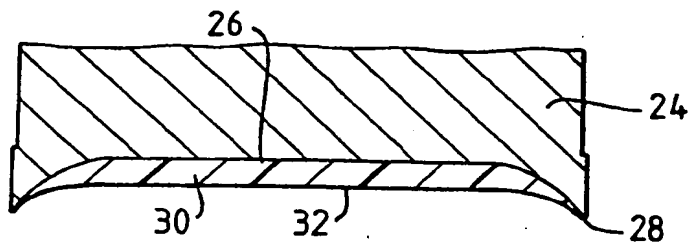


Fig. 5.



PROCESS FOR MAKING SHAPED ARTICLES

This invention is concerned with the production of shaped
5 articles by the compaction of particles. Significant
forms of the invention are concerned with the production
of built detergent products suitable for the laundering
of fabrics and in some cases for other uses such as the
cleaning of hard surfaces, these products being made in
10 the form of shaped solid articles.

One known category of detergent product in the form of a
shaped solid article is a non-soap detergent tablet which
is added to a washing machine at the start of the wash as
15 an alternative to using a powdered or a liquid detergent
composition. Such tablets are formulated for a solution-
washing process with the intention that the soluble
components of the tablet will dissolve rapidly and that
the tablet will break up completely in the wash water
20 giving a wash liquor whose composition is similar to that
which would be given by the use of a detergent powder or
liquid.

Another non-soap detergent product in shaped solid form
25 is a detergent bar used for the handwashing of fabrics
and sometimes also used for cleaning fixed surfaces.
Such bars are used by rubbing on the fabric or surface.
A layer of the bar wears away (e.g. is transferred to the

fabric or the wash liquor) but after use the remainder of the bar can be set aside and stored for use on a future occasion.

- 5 Detergent tablets intended to break up when placed in a wash liquor are normally produced by stamping a particulate detergent mixture so as to compact it from its powder form into a shaped solid article.
- 10 Detergent bars for handwashing have traditionally been produced by the preparation of a doughy mixture which is extruded and allowed to cool into a hard bar. Our UK patent application which is due to be published as UK-A-2260989 discloses that such reusable bars can also be
- 15 manufactured by stamping a particulate composition to compact it into shaped solid form. This is done using a higher compaction pressure than is utilised to form a detergent tablet, hence obtaining a less porous bar which retains its integrity when in contact with water.
- 20
- The stamping of a particulate detergent composition into a shaped solid form serves both to create the shape of an article and increase the bulk density from that of the particulate composition. Such stamping is normally
- 25 carried out using steel dies or punches in contact with the detergent composition. We have found that some detergent compositions stick to the dies, leading to imperfections in the solid articles which are produced by

stamping and/or interruptions in production.

There have been proposals to stamp preformed blanks or billets of soap or certain non-soap detergent

- 5 compositions using dies which are surfaced with a plastic or elastomeric material so that the die surface which comes into contact with a detergent composition has some elasticity.
- 10 Our European patent 276 971B discusses several such prior disclosures and points out that in them the lowest modulus of elasticity which is quoted is 1.9×10^4 pounds per square inch which is equal to 1.3×10^8 Newtons per square metre (Nm^{-2}). 1 Newton per square metre is also
- 15 referred to as 1 Pascal.

In our above-mentioned EP 276 971B it is disclosed that certain detergent compositions can advantageously be stamped into bar form using dies with a total modulus of

20 elasticity lying in a range less than the values quoted in the earlier documents. This prior patent describes this in the context of soap bar compositions and analogous compositions where stamping is applied to previously form billets where a very high proportion of

25 the composition is provided by the soap or non-soap detergent, free fatty acid which serves for superfatting of the composition, and moisture. A concern in the stamping of such compositions is stated to be to maintain

a smooth, glossy surface.

These prior disclosures do not mention any compositions having a rheology other than that of soap and soap mixtures, for which the stamping operation would be carried out on previously extruded billets.

When a particulate composition is compacted, the resulting shaped product does not necessary have the smooth glossy surface aimed at by the prior disclosure in our EP-A-276971B, but instead may have a grainy appearance in which the outlines of the original particles can still be seen.

Other contexts in which particulate compositions are compacted into shaped solid articles are the general technology of tablet formation such as the manufacture of medicinal tablets of various kinds. Problems of the mixture adhering to the dies are known in this field and the standard technique for dealing with such a problem is either to coat the dies with a low energy, "non-stick" surface such as a thin coating of polytetrafluoroethylene or to use lubricants such as magnesium stearate or alternatively to spray the heads periodically with silicones.

In contrast to the prior disclosures which teach stamping using dies having a modulus of elasticity of at least 1.3

x 10^6 Newtons per square metre, we have found that it is advantageous to use dies having a lower modulus of elasticity when stamping shaped solid articles from particulate compositions. Especially, we have found this to be advantageous when stamping shaped solid detergent products from particulate compositions containing inorganic solids or certain crystalline organic builder salts.

Accordingly, in a first aspect this invention provides a process for the manufacture of articles in shaped solid form by compacting a particulate composition in a mould consisting of a plurality of mould parts which are movable relative to each other, characterised in that at least some of the mould parts have, over at least some of their surface area which contacts the product, a total modulus of elasticity within the range from 10^5 up to $8 \times 10^7 \text{ Nm}^{-2}$, preferably less than $7 \times 10^7 \text{ Nm}^{-2}$ and more preferably not over lower values such as $6 \times 10^7 \text{ Nm}^{-2}$, $5 \times 10^7 \text{ Nm}^{-2}$ or even $4 \times 10^7 \text{ N.m}^{-2}$.

We have found that the use of elastic mould parts in accordance with this invention assists in separation of the stamped product from the mould parts, even though the composition which is compacted is very different in character from the compositions contemplated by our EP 276 971B. If the particles are of sufficient size to give a grainy appearance to the stamped product, the use

of elastic mould parts may retain and even enhance this.

For some formulations the total modulus of elasticity may be close to the upper end of the quoted range while for
5 other formulations it may be very beneficial to use mould parts giving a lower total modulus of elasticity.

The particulate composition which is compacted may be a mixture of particles of individual ingredients, or may
10 comprise particles which themselves contain a mixture of ingredients. Such particles containing a mixture of ingredients may be produced by a granulation process and may be used alone or together with particles of single ingredients.

15

A conventional combination of mould parts, to which this invention may be applied, comprises one or more mould parts defining a cavity to receive the particulate composition and a mould part which is movable into the
20 cavity to compact the particles therein.

25

The mould parts which define a cavity preferably comprise a sleeve which is open at both ends, and a part which blocks the sleeve to form a closed-ended cavity.

When an article is stamped in such a cavity the sleeve forms the sides of the article while the part which blocks the sleeve and the part which is movable into the

cavity form opposite faces of the shaped article. After stamping the article the part which blocked the sleeve may slide within the sleeve to eject the article from it.

- 5 It is desirable that at least one mould part comprises a non-elastomeric portion and an elastomeric portion attached thereto at a position to contact the composition during stamping.
- 10 In the arrangement described above it is preferred to employ a sleeve which is not elastomeric, while the part which blocks the sleeve and the part which moves into the sleeve each comprises a non-elastomeric portion and an elastomeric portion attached thereto at a position to
15 contact the composition during stamping.

The term "total modulus of elasticity" used herein refers to the modulus of elasticity measured by compressing the surface which contacts the particulate composition. If a
20 mould part is made of an elastomeric portion overlying a non-elastomeric portion, the total modulus of elasticity will be affected by the presence of the non-elastomeric portion beneath the elastomeric material. It will also be affected by the modulus of the elastomeric material
25 itself, and the thickness of the elastomeric material.

When an elastomeric portion overlies a non-elastomeric portion it will generally be desirable that the

elastomeric material per se has a modulus of elasticity in the range from 5×10^5 to 3×10^7 N.m⁻², better up to 2.5×10^7 N.m⁻².

- 5 It is often desired to stamp a logo or emblem into the surface of a solid article while compacting it. In this invention the shape of the logo or emblem may be formed in the elastomeric portion at the surface of a mould part while a non-elastomeric portion beneath merely has a
10 plain, smooth surface.

- When a mould part has both non-elastomeric and elastomeric portions it may be preferred that only elastomeric material contacts the particulate composition
15 during stamping. However, it may alternatively be preferred that some, preferably only a small area, of the non-elastomeric portion contacts the composition during stamping.

- 20 Each elastomeric part may vary in thickness, for example by tapering at its edges. Preferably it will have a thickness which does not exceed 10 mm, better does not exceed 7 mm, over the whole of its surface area while having a thickness of at least 0.5 mm over some,
25 preferably most, of its surface area which contacts the composition during stamping. A logo or emblem may give rise to a small area of thickness exceeding 10 mm, or, as the case may be, 7 mm.

The non-elastomeric portion of a mould part is suitably made of metal or other rigid material. The elastomeric portion of a mould part can be made from any suitable elastomer. Numerous types of elastomer are available, including thermoplastic, chemically-cured thermosetting and heat-cured thermosetting types. We presently prefer elastomers selected from natural rubbers, silicone rubbers, polyurethanes, nitrile rubbers and butyl rubbers. Use of a heat-cured elastomer may be preferred.

10

A very significant form of this invention is its application to the production of built detergent products. Accordingly, in a further aspect this invention provides a process for the manufacture of a built detergent product in shaped solid form and comprising:

- 5-60 wt% of non-soap detergent active,
 - 10-70 wt% of detergency builder which is a solid selected from water-insoluble inorganic detergency builders, water-soluble inorganic detergency builder salts and water-soluble organic detergency builder salts containing not more than 6 carbon atoms,
 - 25-85 wt% of other materials,
- said process comprising mixing the above ingredients to form a particulate composition and then compacting that composition in a mould consisting of a plurality of mould parts which are movable relative to each other, characterised in that at least some of the mould parts

25

have, over at least part of their surface area which contacts the detergent product, a total modulus of elasticity within the range from 10^5 up to $8 \times 10^7 \text{ Nm}^{-2}$, preferably less than $7 \times 10^7 \text{ Nm}^{-2}$ or even less, such as
5 not more than 6, 5 or $4 \times 10^7 \text{ Nm}^{-2}$.

It will be noted that such products include a detergency builder material which is either inorganic or is an organic salt with at most a short carbon chain, e.g.
10 citric acid. Thus these compositions are different in character and rheology from the compositions disclosed in the earlier disclosure EP-276971B.

One form of these products, as already mentioned, is
15 detergent tablets intended to provide a single does of detergent composition for use in machine washing.

The amount of detergent active in such tablets will generally lie in a range from 5 to 45% by weight of the
20 tablets, possibly at least 10%, possibly also not more than 35% by weight.

The amount of detergency builder will preferably be at least 20% by weight and may possibly lie in a range from
25 25 to 60% or even 50% by weight.

Such tablets will frequently include a peroxygen bleach, such as alkali metal perborate or percarbonate, in

amounts from 2 to 30%, preferably 5 to 25% by weight. The peroxygen bleach is preferably accompanied by an organic bleach precursor which reacts with the bleach to form an organic peroxyacid. A well known example of such a precursor is tetraacetyl ethylene diamine (TAED).
5 Bleach precursor may typically be present as 1 to 10% by weight of the tablet.

The ingredients of a tablet may be wholly water soluble, if a water-soluble detergency builder such as sodium tripolyphosphate is used. Insoluble material may alternatively be included - a likely instance is when the detergency builder comprises an aluminosilicate ion-exchange builder such as zeolite.
10

15

In order that detergent tablets will dissolve and/or break up when placed in water, they should not be compacted with too much pressure. A pressure of not more than $5 \times 10^6 \text{ Nm}^{-2}$ will usually be appropriate.

20

Another form of product within this invention is, as mentioned, a detergent bar for handwashing of fabrics. Such bars are generally formulated to contain 8 to 60%, more often 8 to 45% by weight of non-soap detergent active, 10 to 70% by weight of water-soluble salts and 10 to 70% by weight of water-insoluble material. The quantity of water-soluble salts will include any water-soluble detergency builder salts. The quantity of water-
25

insoluble material will include any water-insoluble detergency builder which is present as well as any quantity of water-insoluble filler materials.

5 Extruded detergent bars used for handwashing in certain countries included insoluble materials, such as talc, kaolin, calcite or bentonite, as a "filler" which although chemically inert is significant in contributing to structural strength and other properties of a
10 detergent bar.

In the bars for handwashing, specified above as a form of this invention, there is both water-soluble and water-insoluble material.

15

If the detergency builder is provided by water-soluble salts, then water-insoluble material present in the bar composition may be the water-insoluble materials traditionally incorporated in laundry detergent bars as a
20 "filler".

If the detergent builder is water-insoluble it will be accompanied in the bar by water-soluble salts, such as sodium carbonate which will govern the alkalinity of the
25 wash liquor in use. An insoluble detergency builder may also be accompanied by the water-insoluble filler materials referred to above.

- A detergent bar will generally contain 10 to 45% by weight of water-soluble builder or will contain 10 to 30% by weight of water-insoluble aluminosilicate builder accompanied by at least 10% by weight of water soluble salts. A bar formulation will usually also contain from 10 to 70% of water-insoluble filler selected from talc, calcite, kaolin, bentonite, finely divided aluminosilicate, silica and calcium silicate.
- 10 The shaped solid products which are produced may be of a traditional bar shape but need not be of such shape. In general the shaped solid products for use in handwashing will be sufficiently large to be handled and will therefore have at least one overall longitudinal dimension of at least 20 mm, and often will be considerably larger, e.g. with at least one overall dimension of at least 50 mm.

- The step of compacting the particles reduces the porosity of the composition. Porosity is conveniently expressed as the percentage of volume which is air. For bars to be used in handwashing it is desirable that the air content of the shaped solid product is less than 30% by volume. The compacting step can be adjusted to produce shaped solids with a range of porosities. Preferably the porosity is not greater than 25% air by volume. A high compaction pressure can reduce porosity to as low as 1%. The preferred porosity is 1 to 25% more preferably 5 to

20% air by volume. A compaction pressure exceeding $5 \times 10^6 \text{ Nm}^{-2}$ will usually be used. Preferred is around $15 \times 10^6 \text{ Nm}^{-2}$.

- 5 The air content of a shaped solid product can be calculated from the volume and weight of the product, provided the air-free density of the solid content is known. The latter can be measured by compressing a sample of the material under vacuum with a very high applied force, then measuring the weight and volume of the resulting solid.

For such bars, which are to be used for handwashing and so are intended to maintain their integrity when brought into contact with water, it is strongly preferred that the particulate composition which is subjected to compaction comprises at least some particles, containing both detergent active and other material, which are formed by granulation; and that the compaction leads to a shaped solid product with an air content less than 30% by volume.

Remaining ingredients which are subsequently mixed with the granulated particles may be additional solid material and/or may be liquid material, although the amount of any liquid materials which is added should be sufficiently limited that the resulting mixture is still particulate.

For bars to be used for handwashing it is preferred that from 40% up to 95% or even 100% of the overall composition is subjected to granulation to form particles containing both detergent active and other materials.

- 5 These particles then preferably contain at least 15%, possibly at least half or even at least 60% of the detergent active in the composition.

A particulate composition intended for compaction into
10 detergent tablets which dissolve and/or break up in water may, optionally, contain such particles formed by granulation, possibly together with particles of single ingredients.

- 15 The granulation procedure may be carried out using a variety of known granulation apparatus. If the formulation to be granulated contains not more than about 20% by weight of liquid or paste-form detergent active, granulation can be carried out with a pan granulator, a
20 Z-blade mixer or a mixer of the type with a rotating inclined bowl provided with scooping plates on its interior (as conventionally used for mixing concrete).

If the formulation to be granulated contains a higher
25 proportion of liquid or paste-form detergent active, it is preferred to employ a high-speed mixer/granulator having both a stirring action and a cutting action. Preferably the stirrer and cutter may be operated

independently of one another and at separately variable speeds. Such a mixer is capable of combining a high energy stirring input with a cutting action, but can also be used to provide other, gentler stirring regimes with or without the cutter in operation. It is thus a highly versatile and flexible piece of apparatus.

A preferred type of batch high-speed mixer/granulator for use in the process of the invention is bowl-shaped and preferably has a substantially vertical stirrer axis. Suitable machines include mixers of the Fukae (Trade Mark) FS-G series manufactured by Fukae Powtech Koygo Co., Japan; this apparatus is essentially in the form of a bowl-shaped vessel accessible via a top port, provided near its base with a stirrer having a substantially vertical axis, and a cutter positioned on a side wall. The stirrer and cutter may be operated independently of one another, and at separately variable speeds.

Similar mixers manufactured in India are the Sapphire (Trade Mark) RMG series of rapid mixer/granulators.

Other similar batch mixers are the Diosna (Trade Mark) V series ex Dierks & Söhne, Germany; the Pharma Matrix (Trade Mark) ex T K Fielder Ltd., England; the Fuji (Trade Mark) VG-C series ex Fuji Sangyo Co., Japan; and the Roto (Trade Mark) ex Zanchetta & Co srl, Italy.

Another mixer found to be suitable is the Lödige (Trade Mark) FM series batch mixer ex Morton Machine Co. Ltd., Scotland. This differs from the mixers mentioned above in that its stirrer has a horizontal axis.

5

The use of such mixers to effect granulation, in particular the Fukae and Lödige mixers, is disclosed in our published pending European applications numbers EP-A-339996, EP-A-340013, EP-A-351937 and EP-A-352135 (Cases
10 C.3236, 3235, 3261 and 3312). As described in those publications, such mixers can be used to carry out granulation of compositions which have previously been spray dried or prepared by mixing (possibly even in the same mixer) and the granulation can increase the bulk
15 density of the particulate composition.

These published applications explain that liquid binder for the granulation may be adequately provided by moisture contained within ingredients of the composition,
20 or by liquid ingredients, making deliberate addition of liquid binder unnecessary.

The amount of liquid binder which is incorporated, including added binder, if any, as well as moisture which
25 is present in solid materials, will typically range from 1 to 10% by weight of the material subjected to granulation. The addition of some liquid binder, generally in an amount not exceeding 6% by weight, may be

desirable.

As described in our above-mentioned published European applications a finely divided particulate material may be
5 added to the mixture. The preferred average particle size of such a material is 0.1-20 micrometers, more preferably 1-10 micrometers.

One example of such a "flow aid" is finely divided
10 amorphous sodium alumino silicate. A suitable material is available from Crosfield Chemicals Ltd, Warrington, Cheshire, England, under the trade mark Alusil. Another possible "flow aid" is finely divided crystalline sodium aluminosilicate.

15

If desired, both crystalline and amorphous sodium aluminosilicates may be used, together or sequentially as flow aids.

20 Other flow aids suitable for use in the process of the invention include precipitated silica, for example, Neosyl (Trade Mark), and precipitated calcium silicate, for example, Microcal (Trade Mark), both available from Crosfield Chemicals Ltd.

25

The amount of flow aid will usually lie in a range from 1 to 12% by weight based on the amount of material subjected to granulation, and often from 2 to 10% by

weight. A preferred amount is from 2 to 6% by weight.

A preferred procedure for the production of granules, which may be in accordance with EP-A-352,135 referred to
5 above, is to fluidise particulate solid alkaline inorganic material, in admixture with other particulate solids, in a mixer/granulator and then add the acid precursor of detergent active to the solid in the mixer/granulator to bring about neutralization of the
10 acid precursor of the detergent in the presence of a solid material and also effect granulation. When carrying out this procedure it may be helpful if a quantity of flow aid material is present with the particulate solids before the addition of the detergent
15 acid commences.

A further quantity of flow aid material may be added after granulation to improve the handling of the particulate material during transfer to the compaction
20 step.

If in-situ neutralisation takes place in the presence of only a low level of moisture, it may not reach completion so that the granules contain some detergent in acid form.
25 However, this should be neutralised rapidly on entering solution during use.

As already mentioned above other ingredients may be

incorporated subsequently to granulating. If these other ingredients are solid they can simply be mixed in, perhaps using the same mixer as carried out granulation (although running at a rather slower speed). If liquid
5 they may be sprayed on but the amount of liquid which is sprayed on should be restricted to the amount which can be absorbed by the solids, so that the composition remains particulate and is not converted into a dough or slurry.

10

It is possible that the mixture which is compacted could itself be produced by an extrusion process of some kind, for example extruding a dough formed by neutralisation so as to give noodles which are then comminuted into small
15 particles.

Detergent actives useable in the present invention may be found in the general classes of anionic, nonionic, amphoteric and zwitterionic actives. Generally anionic
20 and/or nonionic detergent actives will be the main non-soap detergent active present. If any amphoteric or zwitterionic detergent actives are present these will typically be in minor amounts relative to the anionic and nonionic detergent actives.

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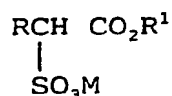
Anionic detergent actives which are particularly envisaged for use in the present invention are alkyl benzene sulphonates of formula



primary alcohol sulphates of formula



- 5 and/or fatty acyl ester sulphonates of formula



in which R is a primary alkyl group containing 18 to 22
10 carbon atoms preferably 10 to 18 carbon atoms, R¹ is a
primary alkyl group containing 1 to 6 carbon atoms
preferably 1 to 4 carbon atoms and M is a cation such
that the detergent active material is water soluble.

- 15 Other anionic detergent actives which may be used include
alkane sulphonates, secondary alcohol sulphates, olefin
sulphonates and ethoxylated alcohol sulphates.

It is possible within the scope of the invention to
20 include soap although in products for hand laundering
soap is preferably absent because it inhibits the
formation of lather by the non-soap detergent active.
Consequently, in such products, it is preferred that if
soap is present at all, the amount of it does not exceed
25 2% by weight of the composition.

Detergent tablets, intended for machine washing are
likely to contain some nonionic detergent active in
addition to anionic detergent active.

Such nonionic detergent active is exemplified by ethoxylated C₈ to C₂₀ alcohols.

5 Fatty acyl alkanolamides may be included in bars for handwashing. They can be useful as lather boosters in such bars.

Products of this invention also include detergency builder and this may be provided by water-soluble salts
10 or by water-insoluble material.

Examples of water-soluble builders are sodium tripolyphosphate, pyrophosphate and orthophosphate; water soluble carbonates, e.g. sodium carbonate; and organic
15 builders containing up to six carbon atoms, e.g. sodium nitrilotriacetate, sodium tartrate, sodium citrate, and trisodium carboxymethyloxysuccinate.

In particular phosphate or polyphosphate detergency
20 builder may provide at least 5% by weight, often at least 10% by weight of the overall composition.

As an alternative to the use of water-soluble builders, the detergency builder may be aluminosilicate ion
25 exchanger, for example zeolite 4A, which is water-insoluble.

Products of this invention may include other detergency

builders in addition to those specified above as essential ingredients. In particular polymeric organic detergency builders such as acrylate/maleate copolymers may be present in conjunction with aluminosilicate ion-exchangers.

Other ingredients may also be present in the overall composition. These include sodium carboxymethyl cellulose, colouring materials, enzymes, fluorescent brighteners, germicides, perfumes and bleaches. Sodium alkaline silicate may be included, although the amount of this or at least the amount added as an aqueous liquid, is preferably restricted so as to keep to a particulate mixture prior to compaction.

15

An embodiment of apparatus for use in the process of this invention will now be described with reference to the accompanying drawings in which:-

- 20 Fig. 1 is a vertical cross-section through a tablet press illustrating its general arrangement;
Figs. 2, 3 and 4 are similar cross-sections showing stages in the cycle of operations of the tablet press; and
25 Fig. 5 is a cross-section through one punch showing the provision of elastomer.

As seen in Fig. 1 the tablet press includes a die 10 in

the form of a sleeve into which fit a lower punch 12 and an upper punch 14. The die 10 defines a cavity 16 closed at its bottom by the lower punch 12. In use a particulate composition is supplied to this cavity by means of a filling shoe 18 which slides on the upper surface 20 of the die.

Initially the filling shoe advances to the position shown in Fig. 2 with the upper punch 14 raised. A particulate composition falls from the filling shoe to fill the cavity 16 above the lower punch 12.

Next as seen as in Fig. 3 the filling shoe withdraws and the upper punch 14 is pressed down into the cavity 16 thus compacting the particulate composition in the cavity to form a tablet. Next, as shown in Fig 4, the upper punch 14 is raised and the lower punch 12 is also raised until the tablet 22 lies at a level with the surface 20 of the die. After this the filling shoe 18 advances, knocking the tablet 22 off the die as it does so while the lower punch descends to the position shown in Fig. 2 for the cycle of operations to be repeated.

Fig. 5 shows a cross-section through one of the punches 12,14. The main portion 24 of the punch is formed of steel. This steel portion is formed with a face 26 which is set back from the rim 28 of the punch, thus forming a cavity in the face of the punch which is partially filled

with a layer 30 of an elastomeric material. The surface 32 of the elastomer contacts the particulate composition during use and thus defines one face of the tablet or bar which is formed in the tableting press. Over most of the area of the punch the thickness of the elastomeric coating 30 is a uniform 4 mm and the total modulus of elasticity observable at the face 32 is in the defined range of 10^5 to 5×10^7 N.m⁻²

10 The coating of elastomer extends over almost the whole surface of the punch which contacts the particulate composition. However, at the rim 28 of the punch the elastomer layer terminates at a point slightly set in from the extremity of the punch thus leaving a small steel rim exposed. This avoids contact between the elastomer 30 and the die 10 through which the punch slides. This is not essential and it would be possible for the elastomer to extend fully to the rim of the punch so that none of the steel portion 24 of the punch contacts the composition during stamping.

20

Both the upper punch 14 and the lower punch 12 are provided with an elastomer-surfaced face which contacts the particulate composition in the cavity 16 as that composition is stamped into a tablet or bar. Thus it is the elastomer which defines the upper and lower surfaces of the bar whereas the side surfaces of the bar are defined by the steel die 10.

25

Example 1

A particulate detergent composition was prepared by granulating the following ingredients in a high speed mixer:

5		
	<u>Ingredient</u>	<u>% by weight</u>
	Sodium linear C11-13 Alkylbenzene sulphonate	24.0
	Sodium tripolyphosphate	20.8
10	Sodium carbonate	10.0
	Calcite	16.12
	Kaolin	16.12
	Minor ingredients	6.46
	Water	6.5

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The granulation was carried out in a Fukae FS30 high speed mixer granulator. Initially the solids were mixed, with agitator speed 300 rpm and chopper speed 3000 rpm. The solids included additional sodium carbonate required for a subsequent neutralisation reaction.

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The agitator speed was reduced to 100 rpm and the detergent was added as alkyl benzene sulphuric acid, over a period of 1 to 2 minutes. A small quantity of water was added, amounting to 2 to 6% of the composition. Neutralisation took place and the mixture formed granules. A small quantity of flow aid (finely divided silica) was added which prevented the granules

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agglomerating into large lumps.

This granular composition was stamped into detergent bars for the handwashing of laundry. When the tableting was
5 carried out using all-steel punches 12,14 in an apparatus as illustrated in Figs. 1-4, the tablets produced stuck to the lower punch 12 and were damaged when ejected from the press.

10 The all-steel punches were replaced with upper and lower punches 12,14 surfaced with an elastomeric material 30 as shown in Fig. 5. In this example the elastomeric material was rubber which on its own displayed a modulus of about $1.4 \times 10^7 \text{ Nm}^{-2}$. The punches each displayed a
15 total modulus of elasticity of $2.1 \pm 0.2 \times 10^7 \text{ Nm}^{-2}$. It was then found possible to make the tablets continuously without the bars sticking to the punch surfaces and suffering damage.

20 The tableting press was modified again by substituting upper and lower punches with a rubber coating shaped as illustrated by Fig. 5 but formed from a harder rubber so that the punches displayed a total modulus of about $7 \times 10^7 \text{ Nm}^{-2}$. The tablets were then observed to stick to
25 the upper punch 14, showing that this rubber/steel composite with hardness greater than that specified for this invention was less satisfactory than the more elastic composite within the scope of the invention.

Example 2

A particulate detergent composition was prepared by granulating the following materials in a high speed mixer:

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	<u>Ingredient</u>	<u>% by weight</u>
	Coconut primary alkyl sulphate	18
	Linear C11-C13 alkyl benzene sulphonate	12
	Sodium tripolyphosphate	15
10	Sodium carbonate	1.5
	Sodium sulphate	16.5
	Calcite	30.85
	Minor ingredients	3.15
	Water	3

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The granulation procedure was similar to that in the previous Example, except that the primary alkyl sulphate was added as its dry sodium salt together with the other solids. At the neutralisation stage slightly more water

20 was added, as additional binder.

This granular composition was formed into detergent bars for handwashing, using apparatus as shown in Figs. 1 to 4. When all-steel punches were used the composition was

25 again found to stick to the punches so that the resulting bars were damaged during ejection from the press.

The press was modified by substituting punches as

illustrated in Fig. 5 having a rubber coating so that the punches displayed a total modulus of elasticity of $2.1 \pm \times 10^7 \text{ Nm}^{-2}$. It was then possible to stamp the detergent bars continuously without them sticking to the punches.

Example 3

A particulate detergent composition was prepared by mixing two preformed granulates containing detergent active and other ingredients, and adding additional ingredients in particulate form. The resulting formulation was:

	<u>Ingredient</u>	<u>% by weight</u>
	Primary alkyl sulphate	5.7
	Ethoxylated alcohol	8.6
10	Zeolite	28.9
	Water soluble organic polymers	9.3
	Sodium carbonate	14.6
	Sodium perborate	14.0
	TAED	7.4
15	Minor ingredients	4.3
	Water	7.2

This mixture was formed into detergent tablets for machine washing, using an Instron universal testing machine to drive a punch into a fixed mould cavity.. When an all-steel punch was used, particles of the composition remained adhering to the punch, and accumulated on it.

When the punch was replaced with a punch which was generally in accordance with Fig. 5, where the total modulus of elasticity at the rubber surface was $1.6 \times 10^7 \text{ Nm}^{-2}$, the accumulation of particles on the punch was significantly reduced.

CLAIMS:

1. A process for the manufacture of articles in shaped solid form by compacting a particulate composition in a
5 mould consisting of a plurality of mould parts which are movable relative to each other, characterised in that at least some of the mould parts have, over at least some of their surface area which contacts the product, a total modulus of elasticity within the range from 10^5 up to $8 \times$
10 10^7 Nm^{-2} .

2. A process for the manufacture of a built detergent product in shaped solid form and comprising:

5-60 wt% of non-soap detergent active,
15 10-70 wt% of detergency builder which is a solid selected from water-insoluble inorganic detergency builders, water-soluble inorganic detergency builder salts and water-soluble organic detergency builder salts containing not more than 6 carbon atoms,
20 25-85 wt% of other materials,
said process comprising mixing the above ingredients to form a particulate composition and then compacting that composition in a mould consisting of a plurality of mould parts which are movable relative to each other,
25 characterised in that at least some of the mould parts have, over at least part of their surface area which contacts the detergent product, a total modulus of elasticity within the range from 10^5 up to $8 \times 10^7 \text{ Nm}^{-2}$.

3. A process according to claim 1 or claim 2 wherein the mould parts comprise one or more mould parts defining a cavity to receive the particulate composition plus a mould part which is movable into the cavity to compact the particles therein.

4. A process according to claim 3 wherein the mould parts which define a cavity comprise a sleeve which is open at both ends and a part which blocks the sleeve.

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5. A process according to any one of the preceding claims wherein at least one mould part comprises a non-elastomeric portion and an elastomeric portion attached thereto at a position to contact the composition during stamping.

6. A process according to claim 4 wherein the sleeve is not elastomeric while the mould part which blocks the sleeve and the mould part which moves into the cavity both comprises a non-elastomeric portion and an elastomeric portion attached thereto at a position to contact the composition during stamping.

7. A process according to claim 5 or claim 6 wherein some of the non-elastomeric portion contacts the composition during stamping.

8. A process according to claim 5, claim 6 or claim 7 wherein each elastomeric portion has a thickness, over at least some of its surface area, which is in a range from 0.5 to 7 mm.

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9. A process according to any one of claims 5 to 8 wherein each elastomeric part is made of a material selected from natural rubbers, silicone rubbers, polyurethanes, nitrile rubbers and butyl rubbers.

10

10. A process according to claim 2 or any one of claims 3 to 9 when appendant thereto wherein the products comprise at least 10% by weight of non-soap detergent active.

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11. A process according to claim 2 or any one of claims 3 to 10 when appendant thereto wherein the products comprise at least 20% by weight of detergency builder.

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12. A process according to claim 2 or any one of claims 3 to 9 when appendant thereto wherein the products contain 8 to 60% by weight of non-soap detergent active, 10 to 70% by weight of water-soluble salts and 10 to 70% by weight of water-insoluble material.

25

13. A process according to claim 2 or any one of claims 3 to 12 when appendant thereto wherein the products comprise at least 25% by weight of materials selected

from sulphite bleaching salt, peroxygen bleaching salt,
bleach activator, inorganic filler and mixtures thereof.

Patents Act 1977
Examiner's report to the Comptroller under
Section 17 (The Search Report)

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(i) UK Cl (Edition L) B5A (AF35H, K, L, AT1P, AT23P, AD20, ANC)

(ii) Int Cl (Edition 5) B28B; C11D; B29C

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T M JAMES

Databases (see over)

(i) UK Patent Office

(ii) ONLINE DATABASES: WPI

Date of Search

30 JUNE 1993

Documents considered relevant following a search in respect of claims

1-13

Category (see over)	Identity of document and relevant passages	Relevant to claim(s)
Y	GB 2260989 A (UNILEVER) - see example 1, pages 17-18 and lines 21-34 of page 21	1-13
Y	GB 1355521 (BRITISH CERAMIC RESEARCH) - see whole document	1-13
Y	GB 0736591 (MICRO PROCESSING) - see page 3, lines 16-47	1-13
Y	EP 0276971 B1 (UNILEVER) - see page 1 line 54, page 2 line 13	1-13



Category	Identity of document and relevant passages 36	Relevant to claim(s)

Categories of documents

- X:** Document indicating lack of novelty or of inventive step.
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P: Document published on or after the declared priority date but before the filing date of the present application.

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Databases: The UK Patent Office database comprises classified collections of GB, EP, WO and US patent specifications as outlined periodically in the Official Journal (Patents). The on-line databases considered for search are also listed periodically in the Official Journal (Patents).

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